(12) UK Patent Application (19) GB (11) 2 189 253 (13) A

(43) Application published 21 Oct 1987

(21) Application No 8709074

(22) Date of filing 15 Apr 1987

(30) Priority data

(31) 8609372 (32) 17 Apr 1986 (33) GB

(51) INT CL[4]
C09K 3/10 C08L 23/16 F16J 15/14 83/07

(52) Domestic classification (Edition I)
C3V BX
C3T 361 501 543 591 KX
C3W 113 208 214 217 303
U1S 1383 C3T C3V

(56) Documents cited
None

(58) Field of search
C3V
C3T

(71) Applicant
Central Electricity Generating Board

(Incorporated in United Kingdom),

Sudbury House, 15 Newgate Street, London EC4A 7AU

(72) Inventor
Stephen Sully

(74) Agent and/or Address for Service
Boult, Wade & Tennant, 27 Furnival Street, London
EC4A 1PQ

(54) Method of sealing joints and jointing compound therefor

(57) A method of sealing opposed surfaces in a joint comprises applying to at least one of the surfaces a heat curable, jointing compound which is non-fluid at rest but flows when sufficient force is applied thereto, bringing the surfaces together, and applying heat to cure the sealant.

The jointing compound comprises a base elastomer (e.g. an EPDM or silicone rubber), a curing agent and a viscosity reductant (e.g. liquid polybutadiene or silicone oil) to reduce the viscosity of the uncured jointing compound, the viscosity modifier becoming chemically bound to the elastomer upon curing of the jointing compound.

Preferably the mating surfaces of the joint are brought together so as to exclude sealant from potential areas of contact and when the joint is between elements of apparatus operated at at least the curing temperature of the jointing compound, the heat required for curing may be provided simply by operating the apparatus at such temperature.

SPECIFICATION

Method of sealing joints and jointing compound therefor

The present invention relates to a method of sealing joints and to jointing compounds. In particular, the invention relates to the use of jointing compounds which can be thermally cured *in situ* to form an elastomeric sealant in the sealing of elements such as flange joiints, for example.

Many techniques are known for sealing elements, for example flange joints, but especially in the case of low differential pressure joints within plant operated at elevated temperature and pressure we have found that the existing techniques are unsatisfactory.

One technique adopted hitherto is to use single-pack room temperature-curing jointing compounds, for example silicones, polysulphides and polyurethanes. Such jointing compounds are difficult to use because they have a very limited handling time and are viscous liquids or pastes. It is difficult to obtain good seals with such jointing compounds and their successful application is a skilled task. Thick applications, as must be used with large gaps, take a long time to cure and until fully cured are liable to be displaced or blown out by the fluid in the system if it is operated, even at low pressure.

Also known are solvent based jointing compounds [e.g. Hylomar (Trade Mark)] and "drying" types (e.g. linseed oil or alkyd based), both of which types are difficult to use because an optimum state of dryness is impossible to achieve on all areas of a large component. The resultant dry materials are hard and brittle and may crack. A further problem with jointing compounds of these types is that they are very difficult to use with large gaps; thick applications take a long time to dry with the consequences mentioned above in connection with room temperature curing jointing compounds and when they dry thick applications can shrink, resulting in leakage. Dried jointing compounds, although exhibiting high initial compression stiffness, undergo subsequent stress relaxation.

A problem generally encountered with polymeric liquid or paste jointing compounds is that they are low molecular weight materials having relatively poor thermal and chemical resistance.

Deformable tapes and cords, for example of PTFE, have also been used to seal mating surfaces, but as with dried jointing compounds they undergo stress relaxation (accelerated by increasing temperature), after an initial period of high compression stiffness.

Instead of a jointing compound, it is known to use gaskets. Stiff gaskets, for example compressed fibre gaskets, cannot conform to uneven surfaces, whilst soft materials (rubber sheet and insertion rubbers) exhibit stress relaxation (accelerated by increasing temperature). Gaskets generally cause separation of the sealing faces and degradation or failure of the gasket results in large leakage because of this separation. This is sometimes also the case with conventional jointing compounds.

US Patent No. 4409056 proposes a curable sealing composition for use in sealing panels of motor cars. The composition is to be applied by caulking gun and is presumably a post-assembly sealant. The viscosity of the composition is adjusted for it to have sufficient flowability at the time of application by controlling the average molecular weight of the copolymer on which the composition is based. This method of viscosity modification is disadvantageous in practice, since great care has to be taken to achieve the desired flowability (manufacture is therefore uneconomic) and it is difficult to provide a range of compositions with different properties suitable for different applications.

Another motor car sealant is disclosed in British Patent No. 1232254. The sealant is to go round windows and windscreens and comprises a synthetic rubber and what is described as a "plasticizer" and is heavily loaded with finely divided iron oxide (an extending filler). In the Examples the minimum quantity of iron oxide used is about 31% by weight of the sealant. The iron oxide is used for dielectric heating of the sealant to cure it *in situ*. The properties of the base rubber are significantly detracted from by components of the composition. The iron oxide would detract from the flowability and strength of the sealant, and in order to enhance the strength of the sealant the inventors include, for example, asbestos fibre which would inhibit flow of the composition. As an alternative to asbestos fibre, the inventors use extremely complex mixtures which include epoxy resin (which would detract from the physical properties of the base rubber) and neoprene, a chlorinated synthetic rubber which would release HCl as it degenerated and would therefore be unacceptable in appreciable quantities in industrial plant or other machinery. The invention taught in GB 1232254 is unsuitable as a basis for a jointing compound for fluid carrying elements of plant or machinery.

A sealant for tanks containing hot liquid is disclosed in GB 989440. The problem addressed by this British patent is the provision of a sealant which can be vulcanised *in situ* under the influence of the hot liquid and which retains enough elasticity in use despite the hot environment. The problem is solved by blending together (1) an elastomer curable at about 100°C or below (e.g. depolymerised natural rubber) and (2) an elastomer which retains its elasticity when subjected to heat (e.g. butyl rubbers, especially chlorinated butyl rubbers). The problem on which our invention is based is entirely unrelated to the problem addressed by GB 989440.

European patent application No. 0065144 is directed towards the provision of a sealant for pneumatic tyres which is tacky when cured and describes a composition comprising (1) a high molecular weight hydrogenated butadiene (the base rubber) and (2) a low molecular weight liquid polymer (the tackifier), the base elastomer providing a crosslinked network in the tackifier. The tackifier would apparently reduce the

viscosity of the uncured system, but this is not referred to in the application which is directed towards the properties of the cured elastomer.

We have realised that it would be desirable to provide a jointing compound suitable for sealing joints in fluid carrying elements of plant or machinery, for example joints in industrial plant or conduits in other apparatus, which is easy to handle or apply, does not cause separation of the sealing faces and is flexible in use. The present invention is based on our appreciation that an elastomer or rubber based jointing compound with suitably modified flow properties could provide such a jointing compound and our further appreciation of a potentially economic and simple way to modify the flow properties in commercially available elastomer or rubber.

In accordance with the present invention there is provided a method of sealing opposed surfaces in a joint, comprising:

applying to at least one of the surfaces a heat curable jointing compound comprising a base elastomer or rubber and a viscosity modifier other than depolymerised natural rubber to reduce the viscosity of the uncured jointing compound, the viscosity modifier being in an amount such that the jointing compound is workable and becoming chemically bound to the elastomer upon curing of the jointing compound, and the jointing compound being non-fluid when at rest but fluid when sufficient force is applied thereto and forming a flexible, non-tacky vulcanisate when cured;

bringing the surfaces together; and

applying heat to cure the jointing compound.

The jointing compound is included within the invention. By "jointing compound" we mean an assembly aid used during assembly of joints and not a post-assembly sealant which has to meet quite different requirements.

The jointing compound will contain curing agent and, preferably, reinforcing filler as conventionally with elastomers and rubbers. By "reinforcing filler" we mean a particulate material which improves the unsatisfactory stiffness of the base rubber or elastomer when cured by a chemical bonding process with the rubber or elastomer. The most common reinforcing elastomer is carbon black, although silica and silicates can be used in some cases, as with silicone elastomers.

As distinct from a reinforcing filler, an "extending filler" (otherwise called an "inert filler") does not chemically interact with the base elastomer or rubber and acts as a diluent and weakens the vulcanisate in one or two important respects.

The jointing compound of the invention does not contain finely divided iron oxide in a significant amount, i.e. an amount sufficient to appreciably detract from the strength of the jointing compound, e.g. does not contain more than 10 wt%, preferably not more than 5 wt% thereof. Similarly, significant amounts of other extending fillers or other inert material are preferably not used in most cases.

The jointing compound should generally not contain appreciable quantities of any material which would detract from the properties of the base rubber, in order that the physico-chemical properties of the jointing compound are not harmed. Thus, the compound should for practical purposes be chemically inert to its environment and not contain anything which would have an appreciable deleterious affect upon flowability in the uncured state, or upon strength or flexibility in the cured state, although the desired properties of the jointing compound will depend on the working environment.

The jointing compounds of the invention are non-fluid when at rest, by which we mean that they do not flow significantly under their own weight during normal sealing operations. The jointing compounds may thus be said to have sufficient inherent viscosity to be inherently non-fluid. However, when sufficient force is applied to the jointing compounds they flow. The inventive jointing compound is thus convenient to use because it can be applied without difficulty to one of the sealing surfaces where it will stay in place until the two sealing surfaces are brought together when sufficient force will be applied for the jointing compound to flow over the sealing surfaces. When the sealing surfaces are once again at rest the jointing compound stays in place in the joint and can be cured. Generally, the jointing compound behaves essentially as a plastic solid during normal application and handling operations.

The required flow properties would be met by a pseudoplastic jointing compound (pseudoplastic behaviour is a reversible decrease in viscosity with increasing shear rate) or by one which exhibits Bingham plastic flow, i.e. which does not flow until a minimum force has been applied and which flows once the minimum force is exceeded.

In the inventive method, the surfaces are preferably brought together so as to substantially exclude the jointing compound from potential areas of contact between the surfaces, i.e. areas which contact each other when the joint surfaces are brought as close together as possible.

The jointing compound is preferably substantially free of volatile substances (e.g. containing not more than 5 wt% volatile components) in order to avoid the problems which might otherwise arise when thick gaps are being sealed (difficulty in achieving dryness throughout the jointing compound and shrinking and cracking).

The jointing compound should generally be readily deformable (i.e. have relatively low inherent viscosity), that is it should flow between the opposed sealing surfaces by the application thereto of only relatively moderate force as would normally be used in the sealing operation, for example, as might be applied manually, e.g. by the manual tightening of nuts on bolts between the two flanges of a flange joint. The jointing compound should thus be free of material which inhibits flow of the compound under

pressure, for example fibre would interfere with flow and the jointing compound should thus be essentially free of fibre.

It is not possible to give an exact definition of an essential range of viscosity for the jointing compounds, but tests indicate that, at a temperature of 20—25°C and a shear rate of 18 l/s, a viscosity of between about 8500 and about 30000 Ns/m^2 is acceptable. A more preferable minimum viscosity at these conditions is about 12500 Ns/m^2, whilst the most preferred range is 12500—20000 Ns/m^2. The skilled man will, in any event, be able to recognise whether a jointing compound has the desired viscosity (deformability).

It is envisaged that, typically, at a shear rate of 73 l/s and a temperature of 20—25°C the viscosity will have reduced to 3500—10500 Ns/m^2, preferably 5000—10500 Ns/m^2, more preferably 5000—8000 Ns/m^2.

The jointing compound must be workable, and in particular must not contain such a large amount of viscosity modifier that the compound loses its normal cohesive, plastic character and ceases to be essentially a plastic solid. It follows that the base elastomer or rubber should not have such a high inherent viscosity that practical levels of viscosity modifier do not sufficiently reduce the viscosity of the base.

The jointing compounds may, as will be appreciated, be relatively easily stored or handled and can be applied to a joint surface without undue difficulty, preferably by pressing a sheet of the material into place or winding cords or strips of the material onto the joint surface, for example. Once applied, the jointing compound will stay in place over the timescale involved in normal sealing operations. However, when jointing compound of the invention has been applied to a mating surface of a joint and the two mating surfaces are brought together, the jointing compound flows under the compressive load applied by the mating faces and may be completely excluded from potential areas of contact between the mating faces. Heat is then applied to the jointing compound to cure it, whereby there may be obtained a uniformly cured layer of jointing compound. If the jointing compound is substantially free of volatile components (e.g. containing not more than 5 wt% volatiles) it is effective even for sealing thick gaps. Usually the jointing compound will be cured at a temperature of at least 100°C and more usually between 120°C and 180°C.

The method of the invention is particularly useful for sealing elements together in apparatus which in normal use operates at an elevated temperature at which the jointing compound will cure. In this case, it is not necessary for heat to be specially applied to the jointing compound because it will be cured during the initial operation of the apparatus after the jointing compound is applied. When this curing technique is adopted, the viscosity of the jointing compound must be sufficient to prevent blow out of the jointing compound prior to curing.

It will be appreciated that, when the jointing compound is excluded from potential sealing face contact areas in a low differential pressure joint, little or no reliance on compressive stress retention is required (there is no load bearing requirement in the jointing compound other than the small seal differential pressure) and the minimum possible leakage area is exposed in the event of seal failure.

The invention may be used in a wide variety of applications, such as in power stations or chemical process plant. We have found the invention particularly useful in the internal sealing of high pressure feed heaters in power stations. Obviously, the jointing compound should not be subjected to high differential pressures which it could not withstand. Of course, the jointing compound will be chosen to be inert to those substances with which it might be contacted and to be stable at the operating temperature of the apparatus concerned. The jointing compound should also be non-corrosive. Thus chlorinated rubbers (e.g. chlorinated butyl rubber) are most preferably not used because they release HCl when they degrade.

The jointing compound comprises a base elastomer or rubber and a viscosity modifier. As the base elastomer any heat curable elastomer may be used so long as the elastomer is resistant to the sealing environment. As an example there may be mentioned EPDM (ethylene propylene diene monomer) rubber as the base elastomer. Other ethylene/propylene rubbers may also be used, or alternatively butyl rubber or polyisobutylene. The base elastomer may comprise more than one type of polymer. The base elastomer may be present in an amount of, typically 40 to 70 wt%, preferably 45 to 65 wt%.

As mentioned above, the base elastomer should not have an excessively high inherent viscosity otherwise unworkably large amounts of viscosity modifier have to be used. As a guide, a Mooney viscosity of 15—30 Units at 120°C will generally be suitable, and more preferably a Mooney viscosity of 18—25 Units may be selected. A conventional fluorocarbon rubber with a Mooney viscosity of 50 Units or more at 100°C has been found to be excessively viscous and, although the chemical resistance of fluorocarbons would be useful in some applications, a lower molecular weight than is customary would have to be used in order to obtain an acceptable viscosity.

When an EPDM rubber is used as the base elastomer and polybutadiene as the viscosity modifier, we have used dicumyl peroxide to effect curing and appropriate curing agents may be easily selected by the skilled person for other systems. Peroxides may be used to cure either unsaturated or saturated systems and are the preferred class of curing agent. The appropriate quantity of curing agent may be readily determined by the skilled person but will often be present in an amount of on the order of 1 to 5% by weight, typically 1.5 to 3 wt%.

The activity of the curing system must be such that the jointing compound can be stored for relatively prolonged periods without curing at ambient temperature and cure at a workable rate at an elevated temperature. Preferably the jointing compound is suitable for curing at a temperature of 100°C or more and

more usually the curing temperature will be from 120°C to, for example, 180°C. The curing period may be from 30 minutes to 8 hours, for example.

The viscosity modifier serves to make the uncured jointing compound flow more readily under load and is a temporary modifier in that it becomes chemically bound with the base material upon curing. Generally, the viscosity modifier is a low viscosity polymer, which, it will be appreciated, has polymerisable sites to allow cross-linking with the base elastomer. The type and amount of viscosity modifier will be chosen in accordance with the base elastomer selected. For example, liquid polybutadiene is preferably used with EPDM rubber in an amount of, for example, from 10 to 25%, more preferably 10 to 20% by weight of the jointing compound. In the case of a silicone base elastomer, the viscosity modifier may be used in an amount of from 5 to 30% by weight of the jointing compound for example, more preferably from 10 to 30%.

The amount of viscosity modifier relative to the amount of base rubber or elastomer will generally be from 5 to 60 parts; more usually 5 to 50 parts, by weight per 100 parts of the base. In the case of EPDM rubber the minimum proportion of viscosity modifier (especially liquid polybutadiene) is preferably 20 parts per 100 parts of base and we have found about 30 parts per hundred parts of base very satisfactory. Of course, the viscosity modifier may comprise a number of different individual components. It is contemplated that the amounts of viscosity modifier used with EPDM rubber apply in general to other hydrocarbon rubbers, especially those listed above.

The viscosity modifier should be distinguished from a conventional plasticiser or process oil which is free in the cured polymer. Such plasticisers or process oils tend to detract from the properties of the elastomer system and should generally not be present in a significant amount. Desirably, any process aid used should crosslink with the other components in the cured jointing compound, for example polyethylene is suitable.

Since the base and the viscosity modifier are to be chemically bound to each other in the cured state, it is preferred that in unsaturated systems both components should have a relatively high degree of unsaturation at which crosslinking may readily take place. For example, it is therefore preferred that the base rubber or elastomer should not be hydrogenated and a vinyl content of 65%—90%, more preferably 70% to 90% is preferred for the viscosity modifier. The cured jointing compound should not be tacky, as this implies relatively high levels of free viscosity modifier.

In one preferred embodiment the base elastomer/viscosity modifier system is a mixture of a base silicone and low viscosity silicone oil.

Typically, the jointing compound will further include a reinforcing filler (e.g. carbon black or silica, the latter being used in silicone based compound) to improve the properties of the jointing compound, for example processability, green strength and vulcanisate strength; a process aid, for example polyethylene (sometimes the process aid may also act as a viscosity modifier); an activator or other co-curing agent e.g. zinc oxide and/or zinc stearate; an accelerator; an antioxidant; and/or a thermal stabiliser, for example. It will be appreciated that in many cases one or more such additives will be necessary to obtain a jointing compound suitable for practical application. However, the jointing compound should not contain significant quantities of fibre, because this will inhibit or prevent flowing of the compound under pressure and granular material will generally be avoided because it will prevent mating surfaces of joints from coming together.

Typically the jointing compound will include the following components in the indicated amounts.

TABLE 1

Ingredient	Amount (wt%)
Base elastomer[a]	40 to 70%
Curing agent	1 to 5%
Process aid	0 to 10%
Reinforcing Filler	15 to 30%
Co-curing agent	3 to 7%
Antioxidant	0 to 2%
Viscosity modifier[b]	5 to 25%

Notes: a. Any of the listed ingredients may comprise a mixture of different substances.
b. Excludes process aid having only a marginal affect on viscosity.

Preferred ingredients and preferred ingredient quantities (especially of the preferred ingredients) are set out in Table 2.

TABLE 2

Ingredient	Amount (wt%)
EPDM Rubber	45 to 65%
Dicumyl peroxide (40% active)	1.5 to 3%
Polyethylene (low molecular weight)	4.5 to 6.5%
Carbon black	15 to 25%
Zinc oxide	2 to 6%
Zinc stearate	0 to 2%
Antioxidant	0 to 2%
Liquid polybutadiene	10 to 20%

Silicone elastomers are available with a sufficiently low viscosity to be used for sealing joints without the addition of a viscosity modifier. Accordingly, the invention includes a method of sealing opposed surfaces in a joint, comprising:

applying to at least one of the surfaces a heat curable jointing compound comprising a silicone elastomer and a curing agent, the jointing compound being non-fluid when at rest but fluid when sufficient force is applied thereto;

bringing the surfaces together; and

applying heat to cure the jointing compound.

The preferred features referred to above in respect of the jointing compound containing viscosity modifier and the method of sealing joints therewith apply also to the method defined in the preceding paragraph.

Table 3 below illustrates a preferred silicone rubber based composition. In this composition, further siloxane is added to the Silastic to reduce the viscosity of the composition by diluting the reinforcing filler:

Ingredient	Amount (wt%)
Silastic HS 30[1]	about 75—90%
Vinyl-substituted polydimethylsiloxane	about 10—30%
Dicumyl peroxide (40% active)	about 1—5%

Notes: 1. Silastic is the trade mark of a silicone rubber composition available from Dow Corning. The composition contains vinylpolydimethylsiloxane and silica as a reinforcing filler.

The jointing compound is prepared by combining the ingredients. For example, all the ingredients except the curing agent may be mixed in an internal mixing machine to prepare a mixture which is then refined, following which the curing agent is dispersed in the mixture on a two roll mill.

In a preferred embodiment, the jointing compound may be taken from the mill as a sheet for subsequent cutting or in bulk for further shaping such as extrusion into cord or strip: the material can be stored flat in the sheet form or coiled on spools with interlayers of non-stick film in the case of cord or strip.

When cured the jointing compound is flexible or elastomeric and can tolerate limited movement. Preferably, the coefficient of thermal expansion of the cured jointing compound is greater than that of the components to which it is applied, which tends to provide an additional sealing force at elevated temperatures.

The invention is described in more detail in the following Examples.

REFERENCE EXAMPLE

The following composition was prepared by mixing the ingredients (without the peroxide) in an internal mixing machine. The mix was then refined and the peroxide dispersed on a two roll mill.

Ingredient	Amount (wt%)
EPDM rubber (Du Pont, Nordel 2522)	59.6%
40% powder dispersion dicumyl peroxide	5.9%
Polyethylene (AC 617A)[1]	5.9%
Carbon black (MT N990)[2]	23.8%
Zinc oxide	3%
Zinc stearate	0.9%
Flectol H (Monsanto antioxidant)	0.9%

Notes: 1. AC 617A is a low molecular weight polyethylene from Allied Chemicals.
The polyethylene is used as a process aid but also marginally reduces viscosity.
2. An ASTM designated grade of carbon black.

This formulation was used in a power station boiler feed water heater, in which the water is at a temperature of 260°C and 2,500 PSI (the differential pressure across the sealed internal parts is very low). The formulation had the desired chemical properties (it was inert) but was too viscous to be a practicable jointing compound.

EXAMPLE 1

A modified version of the composition of the Reference Example was prepared as described in the Reference Example. The modified composition includes liquid polybutadiene to reduce its viscosity.

Ingredient	Amount (wt%)
Nordel 1320[1]	49.3%
40% powder dispersion dicumyl peroxide	4.9%
Polyethylene (AC 617A)	4.9%
Carbon black (MT N990)	19.8%
Zinc oxide	4.9%
Zinc stearate	0.7%
Flectol H (Monsanto antioxidant)	0.7%
Ricon 151[2]	14.8%

Notes: 1. Nordel 1320 is the trade mark of an EPDM rubber sold by DuPont and has a Mooney viscosity of 20 Units at 120°C and a typical number of unsaturated sites for an EPDM rubber.
2. Ricon 151 is the trade mark of a liquid polybutadiene with a vinyl content of 70% sold by Colorado Chemicals. Ricon 151 is being withdrawn from the market and may be substituted by Ricon 150.

EXAMPLE 2

Viscosity characterisation of three jointing compounds

1. *Materials*

Three compositions were mixed using a range of viscosity modifier levels (Ricon 151) to vary the

viscosity of the composition. These compositions were as detailed below, the amounts being in weight percent:

Ingredients	A	B	C
EPDM rubber (Du Pont, Nordel 1320)	57.8	49.3	42.9
Zinc Oxide	5.8	4.9	4.3
Carbon Black MT 990	23.1	19.7	17.2
Zinc Stearate	0.85	0.75	0.65
Polyethylene (AC 617A)	5.8	4.9	4.3
Flectol H	0.85	0.75	0.65
Dicup 40C*	5.8	4.9	4.3
Ricon 151	0.0	14.8	25.7

Note: * A 40% dispersion of dicumyl peroxide on $CaCO_3$ sold by Hercules Powder Co, Inc, Wilmington, Delaware, U.S.A.

All the compositions were initially mixed with an internal mixer (Model B Banbury). A and B mixed successfully but C had to be mixed on a two-roll mill. This was due to the very high loading of Ricon lubricating the mix in the Banbury and stopping effective mixing. When mixed on a 2-roll mill C had the appearance and consistency of a thick putty.

All three mixes were then extruded on a Barwell ram extruder to provide a preform for a Davenport Extrusion Rheometer. All mixes extruded successfully but C tended to slump under its own weight on storage. The Ricon also bled to the surface of C making the material sticky to handle.

2. *Characterisation — test procedure*

The viscosity was measured on a Davenport Capillary Viscometer. This consists of a barrell 19 mm in diameter and 196 mm long with a die of 2 mm diameter and 20 mm long. The rubber was inserted at room temperature into the barrel, in the form of a Barwelled rod.

Measurements were made by driving the piston down at several preset speeds, covering a range of shear rates from 10—1300 l/s. At each speed a pressure reading was taken using a transducer in the side of the barrel wall.

From the pressure reading and the speed the apparent viscosity η was calculated from

$$\eta = \frac{\tau}{G}$$

where

τ = stress and

G = apparent shear rate

Shear rate is at the wall and assumes a parabolic velocity profile which implies Newtonian behaviour.

$$G = \frac{4Q}{\pi R^3}$$

Q = volume rate of flow which one gets from piston diameter and speed

R = radius of die diameter

τ is calculated from pressure

$$\tau = \frac{Rp}{2L}$$

p = presure drop

L = die length

3. *Results*

Viscosity (Ns/m2)

	A	B	C
Temperature (°C)	22	21	22
Shear Rate (l/s):			
18	26909	16183	8866
36	15659	10196	5886
73	9559	6476	3946
181	6079	3944	2440
363	3997	2984	1591
726	2362	1808	1126
1270	—	—	876

It will be noted that compositions B and C are considerably more flowable than the conventional uncured rubber material of composition A.

CLAIMS

1. A method of sealing opposed surfaces in a joint, comprising:

applying to at least one of the surfaces a heat curable jointing compound comprising a base elastomer or rubber, a curing agent and a viscosity modifier other than depolymerised natural rubber to reduce the viscosity of the uncured jointing compound, the viscosity modifier being in an amount such that the jointing compound is workable and becoming chemically bound to the elastomer upon curing of the jointing compound, and the jointing compound being non-fluid when at rest but fluid when sufficient force is applied thereto, forming a flexible non-tacky vulcanisate when cured and not containing finely divided iron oxide in an amount sufficient to appreciably detract from the strength of the jointing compound;

bringing the surfaces together; and

applying heat to cure the jointing compound.

2. A method as claimed in claim 1, wherein the surfaces are brought together so as to substantially exclude the jointing compound from potential areas of contact between the surfaces.

3. A method as claimed in claim 1 or claim 2, wherein the joint is between elements of apparatus operated when in use at at least the curing temperature of the jointing compound and the heat is applied by operating the apparatus.

4. A method as claimed in any one of the preceding claims, wherein the viscosity modifier is a low molecular weight polymer.

5. A method as claimed in any one of the preceding claims wherein the jointing compound has a viscosity of between 8,500 and 30,000 Ns/m^2 at a shear rate of 18 l/s and a temperature of 20—25°C.

6. A method as claimed in claim 5, wherein the viscosity is of between 12,500 and 30,000 NS/m^2.

7. A method as claimed in any one of the preceding claims wherein the base elastomer or rubber is an EPDM rubber or a silicone rubber and the viscosity modifier is liquid polybutadiene or low viscosity silicone oil, respectively.

8. A method as claimed in any one of the preceding claims wherein the joint is a low differential pressure joint subject in use to elevated environmental pressure and temperature.

9. A method as claimed in any one of the preceding claims, wherein the jointing compound comprises from 40 to 70 wt% base elastomer or rubber and from 10 to 25 wt% viscosity modifier.

10. A method as claimed in any one of the preceding claims wherein the jointing compound contains a reinforcing filler.

11. A method of sealing opposed surfaces in a joint, comprising applying to at least one of the surfaces a heat curable jointing compound, bringing the surfaces together, and applying heat to cure the jointing compound, the jointing compound comprising a base EPDM rubber in an amount of from 40 to 70 wt%, a peroxide curing agent, a reinforcing filler in an amount of from 15 to 30 wt% and a liquid polybutadiene viscosity modifier in an amount of from 10 to 25 wt%, and being substantially free of inert filler.

12. A heat curable jointing compound comprising a base elastomer or rubber, a curing agent and a viscosity modifier other than depolymerised natural rubber to reduce the viscosity of the uncured jointing compound, the viscosity modifier being in an amount such that the jointing compound is workable and becoming chemically bound to the elastomer upon curing of the jointing compound, and the jointing

compound being non-fluid when at rest but fluid when sufficient force is applied thereto, forming a flexible, non-tacky vulcanisate when cured and not containing finely divided iron oxide in an amount sufficient to appreciably detract from the strength of the jointing compound.

13. A jointing compound as claimed in claim 12, wherein the viscosity modifier is a low molecular weight polymer.

14. A jointing compound as claimed in claim 12 or claim 13 which has a viscosity of between 8,500 and 30,000 Ns/m^2 at a shear rate of 18 l/s and a temperature of 20—25°C.

15. A jointing compound as claimed in claim 14 which has a viscosity of between 12,500 and 30,000 Ns/m^2.

16. A jointing compound as claimed in any one of claims 12 to 15 wherein the base elastomer or rubber is an EPDM rubber or a silicone rubber and the viscosity modifier is liquid polybutadiene or low viscosity silicone oil, respectively.

17. A jointing compound as claimed in any one of claims 12 to 16 which comprises from 40 to 70 wt% base elastomer and from 4 to 25 wt% viscosity modifier.

18. A jointing compound as claimed in any one of claims 12 to 17 which further comprises a reinforcing filler.

19. A heat curable jointing compound comprising a base EPDM rubber in an amount of from 40 to 70 wt%, a peroxide curing agent, a reinforcing filler in an amount of from 15 to 30 wt% and liquid polybutadiene viscosity modifier in an amount of from 10 to 25 wt%, and being substantially free of inert filler.

20. A method of sealing opposed surfaces in a joint, comprising:

applying to at least one of the surfaces a heat curable jointing compound comprising a silicone elastomer or rubber and a curing agent, the jointing compound being non-fluid when at rest but fluid when sufficient force is applied thereto;

bringing the surfaces together; and

applying heat to cure the jointing compound.

21. A method as claimed in claim 20 and as further defined in any one or more of claims 2, 3, 5, 6, 8, 9 or 10.

22. The use as a jointing compound of a heat curable composition comprising a silicone elastomer or rubber and a curing agent, the jointing compound being non-fluid when at rest but fluid when sufficient force is applied thereto.

23. A method of sealing opposed surfaces with a jointing compound as defined in claim 11 and substantially as hereinbefore described.

24. A jointing compound as claimed in claim 1 and substantially as hereinbefore described.

25. A jointing compound substantially as hereinbefore described in any one of the Examples.

26. A method of reducing the viscosity of uncured elastomeric jointing compound, comprising incorporating in the jointing compound a viscosity modifier in an amount sufficient to reduce the viscosity of the jointing compound to a desired viscosity at which the jointing compound will flow when the mating surfaces of a joint are brought together in the normal way with the jointing compound therebetween but not so large that the jointing compound flows when at rest or ceases to be workable, the viscosity modifier being such that it becomes chemically bound to the elastomer upon curing of the jointing compound and not being depolymerised natural rubber, and the jointing compound forming a flexible, non-tacky vulcanisate when cured and not containing finely divided iron oxide in an amount sufficient to appreciably detract from the strength of the jointing compound.

Printed for Her Majesty's Stationery Office by Courier Press, Leamington Spa, 10/87. Demand No. 8991685.
Published by the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.